Method for control gas diffusion and bubbles formation in liquid porosimetry

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Abstract

The main problem in liquid porosimetry, which prevents to see the pore sizes smaller than 2 microns in diameter, is direct gas diffusion flow through a micro-porous membrane. This diffusion causes bubbles formation below the membrane and that spoils extrusion (intrusion) data, as one cannot distinguish the volume of extrusion (intrusion) liquid from the volume of formatted bubbles. The suggested below method cures the problem by creating the liquid flow below the membrane. The flow washes out all of the small bubbles preventing them to grow. That allows using the membrane at higher differential pressures, even higher than minimum bubble point of the membrane, without spoiling data.

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Figure 1: Basic arrangement for liquid porosmetry.

It can be used in both liquid extrusion porosimetry (LEP) and liquid intrusion porosimetry (LIP). For the LEP mode a presaturated sample is placed on a micro-porous membrane (membrane is supported by a rigid porous plate). The gas pressure is increased in steps and that causes liquid to extrude from the pores. The largest pores extrude first. The top-loading balance for each pressure step measures the liquid out-coming from the sample. The final data for analyzes could be represented as a function relation $V(DP)$, i.e. extruded volume versus differential pressure ($DP = P1 - P2$). Assuming that all of the through-pores are cylindrical we can apply Laplas equation $d = 4g\cos(q)/DP$ to convert the final data to the form $V(d)$.

The same principal works for LIP mode, with the difference in starting at high pressure and decreasing it stepwise; and, of course, the sample is not initially saturated. Also LIP test can be run just after LEP test to see for example liquid extrusion-intrusion hysteresis. Different liquids can be used in LEP and LIP tests. The only requirements are the following: liquid must wet the sample and membrane; the contact angle for the system of sample-liquid-gas must be known; liquid should be stable and should have relatively low viscosity. That is the principle of a liquid porosimetry test.